

# **SYNTHETIC THERMOPLASTIC COMPOSITION, ARTICLES MADE THEREFROM AND METHOD OF MANUFACTURE**

## **CROSS REFERENCE TO RELATED APPLICATION**

5           This application is a continuation-in-part of application Serial Number  
09/322,211 filed May 23, 1999 for Synthetic Thermoplastic Compositions, Articles  
Made Therefrom And Method of Manufacture.

## **BACKGROUND OF THE INVENTION**

### **1. Field of the Invention**

10           The present invention relates generally to improvements in synthetic  
thermoplastic compositions, articles made from such synthetic thermoplastic  
compositions and methods of making thermoplastic articles.

### **2. Description of Related Art**

15           Plastics are moldable chemically-fabricated (synthetic) materials derived mostly  
from fossil fuels, such as oil, coal or natural gas. The long molecules in plastics are  
composed of carbon atoms linked into chains. One type of plastic, polyethylene, is  
composed of extremely long molecules, each containing over 200,000 carbon atoms.  
These long molecule chains give plastics unique properties and distinguish plastics  
from materials such as metals that have crystalline structures. Fossil fuels contain  
20           hydrocarbons, which provide the building blocks for long polymer molecules. The  
building blocks called monomers link together to form long carbon chains called

polymers. The process of forming these long molecules from hydrocarbons is called polymerization. The molecules typically form viscous sticky substances known as resins which are the materials used to make plastic products or articles by heating the resins to their specific melting range and molding them into articles by various methods.

The carbon backbone of polymer molecules often bond with smaller side chains consisting of other elements, including chlorine, fluorine, nitrogen and silicon, for example. These side chains give plastics distinguishing characteristics. For example, when chlorine atoms substitute for hydrogen atoms along the carbon chain, the result is polyvinyl chloride, one of the most versatile and widely used plastics in the world. The addition of chlorine makes this plastic harder and more resistant. The advantages and disadvantages of different plastics are associated with the unique chemistry of each plastic which determines the physical, mechanical and thermal properties of the molded article.

All plastics can be basically divided into two groups: thermoplastic and thermosetting plastic. The two groups differ in the way that each responds to heat. Thermoplastics can be repeatedly softened by heating, and hardened by cooling. Thermosetting plastics harden permanently after being heated once. The present invention is concerned with the thermoplastic family of plastics.

Examples of commonly used thermoplastics are: polyethylene (PE), polyvinyl chloride (PVC), polypropylene (PP), polystyrene (PS), polyethylene terephthalate (PET), acrylonitrile butadiene styrene (ABS), polymethyl methacrylate (PMMA),

polyamide (PA) and polycarbonate (PC). In addition, many variations and hybrid engineered resins based on these are in use today.

The process of forming plastic resins into products is the basis of the plastics industry. Many different processes can be used to make products from thermoplastics.

5 Some of the more common of these processes are injection molding, extrusion molding, blow molding, injection blow molding, blow film extrusion, calendaring, thermoforming, casting and expansion processes. In all these processes, the plastic must be softened or sufficiently liquefied in order to allow the resin to flow and create the shape of the article. For convenience, all these plastic resin forming processes will  
10 be simply referred to as “a molding process” hereinafter.

Because of the importance of thermoplastics to the production of consumer products, and the great number of consumer products made from thermoplastics, the industry is continually searching for ways to improve both the plastic resin systems, and the manner in which an article is made. Specifically, the plastics industry wants to  
15 create articles that have specified chemical and physical strengths, that have better color dispersion, and have improved surface smoothness and texture. The industry is also constantly searching for molding process improvements that reduce:

- (a) cycle time, the amount of time needed to produce each article;
- (b) cure time, the amount of time needed after molding for the article to  
20 be sufficiently hard to be handled;
- (c) energy consumption per article, and
- (d) operating temperature, for both energy conservation and prevention of polymer degradation.

### SUMMARY OF THE INVENTION

The present invention is: (a) a composition of thermoplastic and a naturally occurring aluminosilicate glass (NOAG), (b) articles made from this composition, and (c) a method of making articles from this composition. The NOAG is added to a thermoplastic resin before molding in a manner that disperses it uniformly throughout the resin. The preferred concentration of NOAG is about 0.1% to 3.0 by weight of the total composition of NOAG and thermoplastic resin. The resin may be virgin, a mixture of virgin and recycled, or a mixture of different thermoplastic resins in virgin or recycled form. The NOAG-thermoplastic composition has been found to increase part production rate from 11% to 78% in a wide range of molding processes, including injection, extrusion, blow, blow film, rotary, and compression molding. The NOAG-thermoplastic composition has been found to reduce the energy required to produce a part. The surface finish of the parts made from the NOAG-thermoplastic resin composition appears more smooth and has less noticeable sink marks than parts made from just virgin resin.

### BRIEF DESCRIPTION OF THE DRAWINGS

The exact nature of this invention, as well as its objects and its advantages, will become readily appreciated upon consideration of the following detailed description in relation to the accompanying drawings, in which like reference numerals designate like parts throughout the figures thereof and wherein:

Figure 1 is a chart showing the processing advantages to using NOAG with polypropylene;

Figure 2 is a chart showing the processing advantages to using NOAG with polyethylene;

Figure 3 is a chart showing the processing advantages to using NOAG with polyethylene;

5        Figure 4 is a chart showing the processing advantages to using NOAG with polyethylene;

Figure 5 is a chart showing the processing advantages of using NOAG with a mixture of thermoplastics including recycled resins;

10       Figure 6 is a chart showing the processing advantages of using NOAG with the thermoplastic LEXAN;

Figure 7 is a chart showing the processing advantages of using NOAG with PVC;

Figure 8 is a chart showing the processing advantages of using NOAG with ABS; and

15       Figure 9 is a chart showing the advantages to articles made from various NOAG-thermoplastic resin compositions;

Figure 10 is a graph showing the effect of glass content in the NOAG on cure time of a NOAG-thermoplastic resin composition;

20       Figure 11 is a graph showing the effect of NOAG loading on cure time with the NOAG particle size at less than 325 mesh;

Figure 12 is a graph showing the effect of NOAG loading on injection speed with NOAG particle size less than 325 mesh;

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Figure 13 is a graph showing the effect of NOAG loading on injection pressure with NOAG particle size at less than 325 mesh;

Figure 14 is a  $C^{13}$  nuclear magnetic resonance spectroscopic analysis of LLDPE compounded with NOAG; and

5        Figure 15 is a differential scanning calorimetry analysis of polypropylene compounded with NOAG.

### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

The present invention is a mixture of a naturally occurring aluminosilicate glass  
10    (NOAG) and a thermoplastic resin. Any one of the many well known and readily available thermoplastic resins may be utilized, chosen on the basis of the physical and mechanical properties desired for the molded plastic article.

The inventors conducted tests with various NOAG-thermoplastic resin compositions using various molding machines. A variety of molding processes were  
15    utilized for these tests based on what manufacturing machines were made available to the inventors. The various NOAG-thermoplastic resin compositions were run on conventional injection molding machines, such as a 700 ton Cincinnati Milacron machine, as well as blow molding, extrusion molding and structural foam machines. The range of articles produced by these molding processes using the NOAG-  
20    thermoplastic resin composition included water buckets, bottles, water faucets, milk crates, pill boxes, plastic film, shipping pallets, and railroad ties.

Figure 1 is a chart illustrating the results of a test using NOAG-polypropylene resin composition to mold an article. Percentage reduction on the Y axis 11 is plotted

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against the variables of weight, shot size, cure time and cycle time of articles being produced, on the X axis 12. The composition utilized for producing the injection molded articles is the polypropylene resin with NOAG at a quantity of 1% by weight of the composition. The NOAG-polypropylene resin composition was used in an

5 injection molding machine to make perfume bottle caps. The articles made from the NOAG-polypropylene resin were compared against a baseline of parts made from the virgin resin alone. As shown in the chart of Figure 1, the test results were: the end product weight 13 was reduced by 5%, the volume of plastic that was needed to make the product, the shot size 15, was reduced by 10%, the amount of time that was  
10 required to cool the product after the material was injected into the mold, the cure time 17, was reduced by 28.6%, and the overall cycle time 19 for making each article was reduced by 47.5%.

As shown in Figure 9, the final articles made from the NOAG-polypropylene composition were observed to have an improvement in texture 23, and surface  
15 smoothness 25, and the color distribution 27 of the articles appeared more even.

Figure 2 is a chart illustrating the results of a test using a NOAG-polyethylene resin composition according to the present invention. The composition was NOAG at a quantity of 1% by weight of the composition added to polyethylene. The inventors observed that the final articles were reduced in weight 13 by about 12% and the overall  
20 cycle time per article 19 made from the NOAG-polyethylene resin was reduced by about 32%. As shown in Figure 9, the articles appeared to have improved surface texture 23, less surface imperfections 25, and better color dispersion 27. In addition,

the articles appeared to have a lower final temperature out of the mold 29, and increased strength 31.

Figure 3 is a chart that illustrates the results of another test run using a NOAG-polyethylene resin composition at a higher NOAG loading. The composition was polyethylene with a quantity of NOAG at 1.5% by weight of the composition. The inventors observed that the final weight 13 of the articles were reduced by about 5%, the volume of resin needed to make the articles, the shot size 15, was reduced 27%, and the overall cycle time 19 for the articles was reduced by 12%.

As shown in Figure 9, the articles made from this NOAG-polyethylene composition with more NOAG appeared to have improved texture 23, improved surface 25, and better color 27 distribution than the articles made from virgin resins. The final temperature of the articles 29 appeared to be reduced and the articles appeared to have increased strength 31.

Figure 4 is a chart that shows the results of another test run using a NOAG-polyethylene resin composition at a still higher NOAG loading. The composition was NOAG at a quantity of 2% by weight of the composition added to a polyethylene resin. The inventors observed that the amount of resin needed to make the articles, the shot size 15, was reduced by about 5%, the cure time 17 for each article was reduced by about 12%, and the cycle time 19 for each article was reduced by 14%.

In addition, the articles (Figure 9) appeared to have improved texture 23, improved surface quality 25, and improved color distribution 27. The final temperature 29 of each article appeared to be reduced, and the strength 31 of each article seemed to have increased.



Figure 5 is a chart that illustrates the results of a test using a mixture of different resins or commingled plastic resins with a quantity of NOAG at 1.5% or 2% by weight of the composition. The mixture of resins used for the test included a mixture of high-density polyethylene in virgin and recycled form, a mixture of high-density polyethylene and polypropylene, and a mixture of high-density polyethylene and ABS. Figure 5 shows that, uniformly for each of the NOAG-commingled resin composition with the NOAG quantity at 1.5% or 2% by weight of the composition, the cycle time 19 for the manufacture of the articles decreased by about 40%.

As shown in Figure 9, the articles produced from the NOAG-mixed resin compositions appeared to have improved texture 23, greater surface smoothness 25, better color distribution 27, and increased strength 31.

Figure 6 is a chart that illustrates the results of a test using LEXAN, an engineered thermoplastic proprietary to G.E. Plastics. The test used a NOAG-LEXAN composition with NOAG at 1.5% and 2% by weight of the composition. The composition was used to produce telephone housings and electronic parts. The test results indicated that shot size 15 for each part was reduced by 5%, cure time 17 for each part was reduced by 48%, and cycle time 19 for each article was reduced by 30%.

As shown in Figure 9, the articles appeared to have a final temperature reduction 29 as it came out of the mold.

Figure 7 is a chart that illustrates the results of tests conducted by the inventors using polyvinyl chloride (PVC) in an extrusion molding process. The test used a NOAG-PVC composition with NOAG at 1% by weight of the composition. The test results show that the overall weight 13 of each product was reduced by 45%. At the

same time, as shown in Figure 9, the strength of the article appeared to have increased.

Figure 8 is a chart that illustrates the results of a test conducted using ABS resin and NOAG at 1.5% by weight of the composition to make sprinkler heads in an injection molding machine. The test results show that the cycle time for each article was reduced by about 17%.

The inventors have continued to test and evaluate NOAG-thermoplastic resin compositions since they observed the unexpected and surprising results shown in Figures 1-9. Experimentation was devised in order to ascertain whether there was an optimized range for the NOAG. More scientific procedures were adopted to prove or disprove the results observed by the earlier test results shown in Figures 1-9.

Naturally occurring aluminosilicate glass (NOAG) is an amorphous material. Since amorphous materials do not have a regular crystalline structure or molecular structure, it is not possible to define its composition by means of a chemical formula. The only way to define the composition of an amorphous material is by chemical analysis. As is customary in the geological sciences, the chemical composition of naturally occurring aluminosilicate glass may be expressed in terms of weight percent of the element oxides.

#### Preferred Chemical Composition of NOAG

As it turns out, naturally occurring aluminosilicate glasses have a large chemical range and form. This is most probably due to the number of different geologic processes that create them. However, the overwhelming majority of natural aluminosilicate glasses form as a result of igneous processes. The composition of

natural aluminosilicate glasses can encompass nearly the entire range of igneous rocks. However, natural glasses are most common and abundant in felsic igneous rocks (e.g., rhyolite or its crystalline equivalent, granite). Even within felsic igneous rocks, there is considerable chemical variation among rocks from different locations. We have  
5 discovered that the NOAG that works best in the creation of our NOAG-thermoplastic resin compositions has the following weight percent of element oxides within the listed chemical range.

10	SiO <sub>2</sub>	66-77%
	TiO <sub>2</sub>	0-2%
	Al <sub>2</sub> O <sub>3</sub>	10-22%
	Fe <sub>2</sub> O <sub>3</sub>	0-4%
	MgO	0-2%
15	CaO	0-3%
	Na <sub>2</sub> O	2-8%
	K <sub>2</sub> O	2-8%
	All other element oxides	0-1%
	H <sub>2</sub> O	0-20%

Aluminosilicate glasses occur naturally when molten silicate rock (magma) is chilled so rapidly that there is insufficient time for the relatively unstructured melt to  
20 form crystalline minerals with a regular internal structure. The chaotic structure of the molten silicate is frozen due to the rapid cooling. Typically, rapid cooling occurs when magma is erupted onto the earth's surface. The eruption mechanism can range from quiescent to highly explosive. The form of the eruptive process may be a quiet lava flow to highly explosive eruptions that give rise to deadly "glowing avalanches" of  
25 essentially molten, but disaggregated magma. Thus, the texture of the aluminosilicate glass may vary from a solid mass, in the case of lava flow, to fine particles of ash resulting from highly explosive eruptions.

All physical forms of the aluminosilicate glass may be used as starting material. In a preferred method of preparation, the glass rock is milled to particle sizes on the order of 100 microns or less. The crushing and milling process destroys all textural features of the original rock (i.e., solid rock or ash particles). Therefore, its original  
5 textural features are irrelevant to the performance of the NOAG in composition. As a result, all naturally occurring aluminosilicate glass within the composition range specified above, regardless of its original texture or geologic mechanism of formation, may be used as a starter material.

Most naturally occurring aluminosilicate glasses contain crystalline minerals as  
10 well. Magma as it usually occurs is more than simply molten silicate rock. Besides molten silicate rock, it may contain bits and pieces of crystalline minerals that were scavenged from the walls of the conduit through which the magma passed, crystalline minerals formed due to cooling of the magma as it passes through the earth's crust (e.g., quartz, feldspar, and biotite), as well as crystalline minerals that can form during  
15 eruption, transport, deposition, and cooling of the molten silicate rock (e.g., cristobalite, feldspar, quartz, and biotite). Furthermore, aluminosilicate glass is inherently unstable and can form crystalline minerals (e.g., cristobalite, feldspar, quartz, smectite-groups clays, kaolinite-group clays) over geologic time with or without the catalytic activity of heat, water or dissolved chemicals.

20 All the crystalline minerals contained in the naturally occurring aluminosilicate glass are considered impurities with respect to the present invention. Through experimentation, the inventors have discovered that the crystalline minerals are not effective components in the thermoplastic molding environment. A series of NOAG-

resin compositions were formulated in which the primary variable was the abundance of glass in the NOAG. The effect of these NOAG compositions was assessed in an injection molding press by determining the change in cure time as a function of glass content. Figure 10 is a graph that shows the results of that test. As curve 33 shows, reduction in cure time is directly proportional to the glass content of NOAG. These results show us that the crystalline minerals actually degrade the performance of the synthetic NOAG-thermoplastic resin composition of the present invention.

#### Preferred NOAG Concentration In The Composition

The concentration of NOAG in the NOAG-thermoplastic resin composition is defined by the weight percent of NOAG in the NOAG-resin composition. The NOAG may be introduced into the resin by dry blending a concentrate pellet of NOAG and a universal carrier such as LLDPE, or by direct compounding into the selected resin.

Unexpected and surprising results occur with unusually low concentrations of NOAG. Although a minimum concentration of NOAG has not been defined, addition of as little as 0.25% by weight, leads to the unexpected reduced cure time of 10-16%. As shown in Figure 11, graph 39 shows NOAG having less than 325 mesh particle size in a carrier reducing cure time by 10% when only 0.25% by weight of the composition is used. Graph 41 of Figure 11 shows directly compounded NOAG having less than 325 mesh size reducing cure time by 16% when only 0.25% by weight of the composition is used. As shown by both graphs 39 and 41, increased concentrations from 0.5% to 1.0% of less than 325 mesh NOAG provides even greater decreases in cure times to a maximum of 28%. The upper limit of NOAG concentration has not

been definitively determined. It is presently preferred that the NOAG concentration be below 3.0% by weight of the NOAG-thermoplastic resin composition.

Application Of The NOAG-Thermoplastic Resin Composition

The NOAG-thermoplastic resin composition discovered by the inventors would be of little interest if there was limited or no application for it. To the contrary, the NOAG-thermoplastic resin composition has an exceptionally wide application, both in terms of resin types and in terms of molding processes.

Of paramount importance is the fact that the NOAG-thermoplastic resin compositions of the present invention are not restricted to one or a few resin types. This has been established by field testing of the NOAG-resin compositions in injection-molding presses making a variety of items and using a variety of thermoplastic resins. Table 1 below demonstrates that the NOAG-thermoplastic resin compositions using a variety of resins are substantially better than virgin resins when judged by cycle time reduction. Reduced cycle time translates to increased throughput on the number of articles that can be produced in a time frame.

TABLE 1

Process	Mach Mfr/Size	Product/Cavities	Material/Cost	Sec.-Original Cycle	Sec/Cycle (-) or fpm (+) Line Speed	Increased Throughput
InjM	Toyo/200	Handle	ABS	34.6	8.4	32.00%
InjM	Penwalt Stokes	Figure	ABS	65	28	76.00%
InjM	Cinc-Mil/300	Sprinkler/4	ABS-Eng	43.5	7	19.18%
InjM	Cinc-Mil/300	Cone	ABS-Eng	34	4	11.76%
InjM	JSW/700	Support	ABS-Eng	279.6	239.6	27.32%

Process	Mach Mfr/Size	Product/Cavities	Material/Cost	Sec.-Original Cycle	Sec/Cycle (-) or fpm (+) Line Speed	Increased Throughput
InjM	Cinc	Hinge Cover/4	Dynaflex	25.5	8.2	47.40%
InjM	Engel/750	Tub	HDPE	62.22	7.78	14.29%
InjM	Cinc-Mil/700	Milk Crate	HDPE	35.5	12	51.08%
InjM	Cinc-Mil/700	Crate	HDPE	18.5	6.5	54.17%
InjM	Mire	Chair	HDPE	48.5	9.5	24.36%
InjM	VanDorn/750	Soda Crate/2	HDPE/LPDE w/slip agent	25.5	10.2	66.67%
InjM	Toyo/200	Water Faucet/12	LDPE	17.9	4.7	35.61%
InjM	VanDorn/170	Tel housing	Lexan w/additive	27	7.2	36.36%
InjM	VanDorn/150	PN-15XXX/2	Lexan H136 w/additive	26.65	10	60.06%
InjM	Arburg	Fitting	Nylon 6/6	11.1	2.2	25.00%
InjM	Sandretto	Housing/16	Nylon 6	17.1	3.5	25.74%
InjM	Cinc-Mil/150	Sprocket/2	Nylon 6/6	39.7	12.1	55.08%
InjM	Penwalt Stokes	Figure	PP	50	17	52.00%
InjM	Mitsubishi/720	Tile	PP	59.85	7.67	14.70%
InjM	Van Dorn/770	Office Part	PP	35.5	9.7	37.60%
InjM	Magna/600	Office Part	PP	28.8	5.7	24.68%
InjM	Olma/550	Battery Case/2	PP	51.6	12.14	30.77%
InjM	Cinc-Mil/750	Shutter	PP	73	28.7	64.79%
InjM	Cinc-Mil/440	Warm Light/2	PP	56.5	13	29.89%
InjM	Nissel/399	Brush handle/10	PP	39.9	11.6	40.99%
InjM	Nissel/399	Brush/4	PP	38.4	16.8	77.78%
InjM	Magna/310	Flower Pot	PP	27.5	9.3	61.10%
InjM	Cinc-Mil/310	Container/8	PP	25.48	4.48	21.33%
InjM	JSW/700	Tray	PP	57.5	11	23.70%
InjM	Cinc-Mil/1000; Maxed	Tote	PPcoP	32.17	5.96	22.74%
InjM	Cinc-Mil/1000	Chair backs	PPcoP	139	25	21.93%





**TABLE 2**

Sample	Wt. % NOAG	Injection Speed inches/seconds	Cure Time in seconds	Cycle Time in seconds
<b>TPO</b>				
Virgin TPO	0.00	6.0	20.0	49.0
TPO + NOAG	1.00	6.0	18.5 (-8%)	34.5 (-26%)
<b>NANOCOMPOSITE</b>				
Virgin Nano	0.00	6.0	30.0	68.6
Nano + NOAG	1.00	6.0	13.0 (-57%)	43.2 (-37%)
<b>POLYPROPYLENE</b>				
Virgin PP	0.00	6.75	34.5	47.8
PP + NOAG	0.25	6.75	31 (-10%)	44.4 (-7%)
PP + NOAG	0.50	6.75	30.0 (-13%)	43.6 (-9%)
PP + NOAG	0.75	6.75	29.0 (-16%)	42.8 (-11%)
PP + NOAG	1.00	6.75	27.0 (-22%)	40.6 (-15%)
PP + NOAG	1.50	6.75	26.5 (-23%)	40.2 (-16%)

Besides testing the NOAG-thermoplastic resin compositions in injection molding systems, other thermoforming environments also were used. The results in these environments are equally unexpected and surprising. In extrusion molding, for example, it was clearly unexpected that a resin containing a solid (NOAG) can be extruded at rates of 34-211% greater than that of the resin alone. Furthermore, extruded articles such as tubing and wire sheaths exhibited a more uniform thickness and better surface finish. In blow molding systems, it was surprising that article throughput increased 17-35% with the addition of a solid (NOAG) to a resin. As illustrated in Table 3 below, significant, unexpected, and surprising results were obtained in increased throughput as measured in feet per minute (fpm) when using NOAG in combination with six different thermoplastic resins.

**TABLE 3**

<b>Molding Process</b>	<b>Molding Equipment</b>	<b>Resin System</b>	<b>Standard Line Speed/Cycle Time</b>	<b>Line Speed/ Cycle Time With NOAG</b>	<b>Increased Throughput</b>
Extrusion	NRM 111	MDPE	11.0 fmp	16.0 fpm*	45.50%
Extrusion	NRM 111	PP	22.0 fpm	29.5 fpm*	34.1%
Extrusion	N/A	PVC	123 fpm	260 fpm	211.4%
Blow Molding	BEKUM H-121	HDPE	14.5 sec.	10.72 sec.	35.3%
Blow Molding	Hayssen	HMPE	67 sec.	57 sec.	17.5%
Blow Molding	Jackson	LDPE	30.5 sec.	25.5 sec.	19.6%

\* blowing agent reduced by 50%

As a general rule, the typical molding temperature of a given virgin resin can be lowered when molding with a NOAG-resin composition and still maintain the ability to mold a high-quality part. In one experiment with PP, the nozzle temperature was lowered from 410°F to 390°F and the barrel temperature was lowered from 400°F to 370°F with no decrease in part quality. Temperature decreases of 20-40°F are typical for many resins in a wide variety of molding environments. A lower barrel and nozzle temperature is important for two reasons: (1) the lower resin temperature in the barrel means that the resin is cooler when it reaches the mold; therefore, the cure time can be reduced significantly, and (2) the lower resin temperature reduces the amount of thermal degradation of the resin, particularly when operating temperatures are near or at the maximum operating temperature for the resin. Reducing thermal degradation is critical to assuring the physical, mechanical and thermal properties of the resin as well as assuring longevity of the molded article.

A NOAG-resin composition also reduces the wear on molding machines. In one experiment on an injection press, the injection speed was set at 2.25 in/sec.

Various NOAG-resin compositions were sequentially introduced to the press to determine how the injection speed would change as a function of NOAG loading. In figure 12, curve 55 teaches us that at NOAG concentrations of 0.5% or greater, the injection speed increases with NOAG loading. Since the power settings for the injection speed were not changed, the increase in injection speed means that the NOAG-resin composition provided less resistance to injection. Another measurement, the pressure in the barrel during injection, also reflected the greater ease in injecting the NOAG-resin composition. As shown by curve 56 on Figure 13, the pressure in the barrel decreased with an increase of NOAG in the NOAG-resin composition. Both of these parameters indicate that a press would require less force and with less force, there would be less wear on the press. Other molding machines most likely have less wear and tear upon introduction of a NOAG-resin composition as well. For example, an extruder screw often increases in speed when a NOAG-resin composition replaces virgin resin.

The conclusion that the NOAG-resin composition requires less force to mold articles is supported by measurements of electrical consumption. In one experiment, PP was injected molded. The press was optimized for virgin PP and the electrical consumption per part was determined to be 0.405 Kwatts/article. Molding the part with the NOAG-resin composition required only 0.391 Kwatts/article or about 3.5% less energy. What is even more surprising and unexpected is the fact that barrel and nozzle temperatures could be reduced 20-30°F and this energy savings as compared to virgin resin was not included in calculation of the 3.5% energy reduction.

NOAG-resin compositions effectively clean the barrel and screw regions of molders. On numerous occasions, we have noted molded or extruded plastic articles of NOAG-resin that are heavily contaminated with bits and pieces of charred resin, colored resin unlike any recently used in the molder and other contaminants such as hairs, paper fiber or unknown materials. In long testing cycles, bits of charred or colored resin occasionally appear some 6 hours after introduction the NOAG-resin composition. In one particularly noteworthy experiment, a series of 6 NOAG-resin samples were progressively compounded in an extruder and then injected molded into test plaques. The objective was to determine whether NOAG-resin compositions with variable NOAG loadings could be as optically clear as virgin resin. The expected result was that the optical clarity would decrease with NOAG loading. In fact, there was no correlation with loading, rather the correlation was with the sequence of extrusion and injection molding—the first samples had the least optical clarity and the last had the best. In fact, the test was such a failure, the molder extruded new compounded NOAG-acrylic pellets and molded new test plaques after cleaning the extruder and injection molder. Although the results were somewhat better, there were still visible bits of charred and colored resin in the clear acrylic, thus indicating that the NOAG-resin composition was better at removing contaminants stuck to the screw and barrel surfaces than even their best cleaning method.

Many resins are manufactured with chemical agents or pigments to improve the moldability of the resin. The NOAG-resin composition enhances the performance of the additives. For example, extruded foam articles require 50% less blowing agent to achieve the same quality part (Table 3). The impact resistance of NOAG-resin

composition in which the resin has a chemical impact modifier is 40% higher than the resin without NOAG. On a more qualitative basis, pigmented plastic articles composed of NOAG and resin have a far more uniform distribution of color, and in some cases, a greater saturation of color than resin alone.

5           As already noted, a particular resin is chosen for the manufacture of an article because of the physical, mechanical and thermal properties it will provide the finished articles. As noted above, the addition of NOAG to thermoplastic resins creates a new composition that has significantly different responses to the heat and pressure typical in a thermoforming environment. These different responses, as already described, are  
10       great attributes of the new NOAG-thermoplastic resin composition. However, these attributes would be of little interest if the synthetic NOAG-thermoplastic resin composition produced articles that had physical, mechanical and thermal properties that are substantially different from articles produced by the virgin resin.

          Extensive evaluation of the physical, mechanical and thermal properties for the  
15       NOAG-thermoplastic resin compositions have failed to identify any consistent substantial difference between articles made from virgin resin and articles made from the NOAG-resin composition. The inventors tested the NOAG-thermoplastic resin composition using six different resins. The NOAG was added at 1% or 1.5% by weight of the composition. The results shown in Table 4 below indicate that there are  
20       no systematic or substantial changes in any of the measured properties across the spectrum of tested resins.

**TABLE 4**

	Polypropylene	Polypropylene plus 1% NOAG	High Density Polyethylene (HDPE)	HDPE plus 1% NOAG
<b>PHYSICAL</b>				
Specific gravity (g/cm <sup>3</sup> )	0.893	0.897	0.934	0.941
Shore hardness (D- scale)	62	62	62	62
<b>MECHANICAL</b>				
Notched Izod impact, 73°F	0.5	0.5	0.5	0.6
Tensile strength @ yield (PSI)	4651	4745	3000	3066
Tensile strength @ peak (PSI)	4656	4751	3005	3070
Tensile strength @ break (PSI)	2622	2872	1381	1070
Tensile elongation @ yield (%)	10.1	10.1	10.5	10.3
Tensile elongation @ break (%)	131.5	157.9	88.2	88.0
Tensile modulus (PSI)	196200	212400	128200	129800
Flexural strength @ yield (PSI)	5672	5782	2682	2797
Flexural strength @ peak (PSI)	5688	5801	2751	2809
Tangent Flexural Modulus (PSI)	206300	201300	128800	136300
<b>THERMAL</b>				
Thermal analysis (DSC), °C (Peak melt temp.)	171.4	169.1	134.5	133.0
Heat deflection @ 264 PSI (°C)	60	59	52	50
Brittleness temperature (-80°C)	-80 (pass) no deformation	-80 (pass) no deformation	-80 (pass) no deformation	-80 (pass) no deformation
	Lexan 121R	Lexan 121R plus 1.5 NOAG	Zytel 101L natural	Zytel plus 1.5% NOAG
<b>PHYSICAL</b>				
Linear mold shrinkage	0.0046	0.0045	0.162	0.129
Water absorption	0.083	0.077	1.00	0.90

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	Lexan 121R	Lexan 121R plus 1.5 NOAG	Zytel 101L natural	Zytel plus 1.5% NOAG
Specific gravity (g/cm <sup>3</sup> )	1.18	1.19	1.12	1.13
<b>MECHANICAL</b>				
Notched Izod impact, 73°F	12.8	11.1	1.2	0.96
Notched Izod impact, -40°F	--	--	0.75	0.73
Tensile strength @ yield (PSI)	8429	8386	9570	9480
Tensile strength @ Peak (PSI)	8501	8462	9583	9576
Tensile strength @ break (PSI)	8032	7704	9574	9571
Tensile elongation @ yield (%)	7.8	7.5	6.8	6.6
Tensile elongation @ break (%)	80.9	74.2	32.6	22.9
Tensile modulus (PSI)	325700	322000	356100	356500
Flexural strength @ yield (PSI)	12901	12934	12896	12913
Flexural strength @ peak (PSI)	12921	12954	12912	12936
Tangent Flexural Modulus (PSI)	354900	344200	317000	350700
<b>THERMAL</b>				
Thermal analysis (DSC), °C (Glass/Peak melt temp.)	147.2	146.3	265.1	267.4
Heat deflection @ 66 PSI (°C)	135	135	211	211
Heat deflection @ 264 PSI (°C)	123	123	79	79
Brittleness temperature (-80°C)	-80 (pass) no deformation	-80 (pass) no deformation	-80 (pass) no deformation	-80 (pass) no deformation
	TPO	TPO plus 1.0% NOAG	Proprietary Nanocomposite	Nanocomposite plus 1.0% NOAG
<b>MECHANICAL</b>				
Notched Izod impact, 73°F	8.2	5.8	7.6	7.6
Notched Izod impact, -40°F	0.73	0.58	0.87	0.84

	<b>TPO</b>	<b>TPO plus 1.0% NOAG</b>	<b>Proprietary Nanocomposite</b>	<b>Nanocomposite plus 1.0% NOAG</b>
Gardner falling dart impact	181	178	178	170
Tensile strength @ yield (PSI)	2890	2830	2990	2930
Tensile strength @ peak (PSI)	2890	2830	2990	2930
Tensile strength @ break (PSI)	2250	2310	2210	2390
Tensile elongation @ yield (%)	13	13	17	16
Tensile elongation @ break (%)	>500	>500	120	370
Tensile modulus (PSI)	10700	10400	11300	11100
Flexural strength @ yield (PSI)	3390	3310	3100	3060
Flexural strength @ peak (PSI)	3680	3600	3320	3280
Tangent Flexural Modulus (PSI)	172000	166000	129000	133000
<b>THERMAL</b>				
Heat deflection @ 66 PSI (°C)	135	135	211	211

Differences between virgin resin and NOAG-thermoplastic resin composition articles were often well within uncertainty of measurement or could not be repeated in successive tests of the same material.

- Of equal significance, the physical, mechanical and thermal properties of the
- 5 NOAG-thermoplastic resin compositions are similar even with variation in the concentration of NOAG at less than 325 mesh particle size as shown by Table 5 below.



**TABLE 5**

	PP+ Carrier	PP	0.50%*	1.00%*	1.50%*	2.00%*	2.50%*	3.00%*
<b>Tensile (psi)</b>								
Tensile @ max	4,640	4,660	4,840	4,860	4,840	4,840	4,650	4,500
Tensile @ break	2,540	1,580	1,960	1,860	1,420	1,450	1,490	350(?)
Tensile @ yield	4,640	4,660	4,840	4,860	4,840	4,840	4,650	4,500
Tensile modulus	16,400	15,800	16,700	16,800	16,600	16,700	16,400	15,800
Elongation @ yield (%)	10	10	10	10	9.4	9.3	9.4	9.0
Elongation @ break (%)	>500	>500	>500	480	>500	>500	>500	>500
<b>Flexural Modulus (psi)</b>								
Flex strength @ yield	5,890	5,830	5,900	5,900	5,860	5,870	5,900	--
Flex strength @ max	6,120	6,090	6,150	6,190	6,110	6,140	6,190	--
Tangent Flex. Modulus	179,000	189,000	197,000	199,000	198,000	198,000	201,000	197,000
<b>Notched Izod (ft-lb-in)</b>								
Energy @ 23°C	0.62	0.64	0.54	0.53	0.50	0.53	0.53	0.52
Energy @ -40	0.37	0.34	0.30	0.30	0.31	0.30	0.30	0.34
<b>Heat deflection temperature (°C)</b>								
@ 66 psi	97	106	103	104	105	106	107	106

\* % of NOAG by weight of the composition.

The physical, mechanical, and thermal properties of articles made from the NOAG-resin composition were found to be comparable for varying concentrations of water in the NOAG as shown in Table 6 below. Water is the only potentially volatile component in NOAG that could react with a resin. No significant variation in physical, mechanical or thermal properties as a function of water content teaches that water does not interact or react with the resin.

**TABLE 6**

	PP + Carrier	PP	100%*	50%*	0%*
<b>Tensile (psi)</b>					
Tensile @ max	4,640	4,660	4,860	4,770	4,730
Tensile @ break	2,540	1,580	1,860	1,580	2,000
Tensile @ yield	4,640	4,660	4,860	4,770	4,730
Tensile modulus	16,400	15,800	16,800	16,600	16,300
Elongation @ yield (%)	10	10	10	10	9.9
Elongation @ break (%)	>500	>500	480	>500	>500
<b>Flexural Modulus (psi)</b>					
Flex strength @ yield	5,890	5,830	5,900	6,050	6,050
Flex strength @ max	6,120	6,090	6,190	6,310	6,250
Tangent Flex. Modulus	179,000	189,000	199,000	199,000	206,000
<b>Notched Izod (ft-lb/in)</b>					
Energy @ 23°C	0.62	0.64	0.53	0.54	0.49
Energy @ -40°C	0.37	0.34	0.30	0.31	0.30
<b>Heat deflection temperature (°C)</b>					
@ 66 psi	97	106	102	106	108

\* Amount of water available at typical molding temperature of 500°F as compared to NOAG as found.

- The physical, mechanical, and thermal properties of articles made from the
- 5 NOAG-resin composition were found to be comparable for various mineral compositions in the NOAG for a NOAG loading of 1% and particle size of less than 325 mesh, as shown in Table 7 below.

**TABLE 7**

<b>Composition</b>	<b>PP + Carrier</b>	<b>PP</b>	<b>95% A 5% B</b>	<b>50% A 50% B</b>	<b>9% A 91% B</b>	<b>86% A 7% B 7% C</b>	<b>68% A 8% B 24% C</b>
<b>Tensile (psi)</b>							
Tensile @ max	4,640	4,660	4,860	4,790	4,770	4,840	4,700
Tensile @ break	2,540	1,580	1,860	1,650	2,930	1,960	2,200
Tensile @ yield	4,640	4,660	4,860	4,790	4,770	4,840	4,700
Tensile modulus	16,400	15,800	16,800	16,600	16,400	16,600	16,200
Elongation @ Yield (%)	10	10	10	9.6	9.5	9.9	9.8
Elongation @ Break (%)	>500	>500	480	>500	>500	>500	>500
<b>Flexural Modulus (psi)</b>							
Flex strength @ yield	5,890	5,830	5,900	6,140	6,020	6,020	5,900
Flex strength @ max	6,120	6,090	6,190	6,410	6,350	6,290	6,160
Tangent Flex. Modulus	179,000	189,000	199,000	185,000	194,000	193,000	189,000
<b>Notched Izod (ft-lb/in)</b>							
Energy @ 23°C	0.62	0.64	0.53	0.53	0.53	0.50	0.58
Energy @ -40°C	0.37	0.34	0.30	0.32	0.31	0.35	0.36
<b>Heat deflection temperature (°C)</b>							
@66 psi	97	106	104	101	105	105	104

A - NOAG

B - Crystalline silicates

C - Clay minerals

- 5 An absence of differences in physical, mechanical and thermal properties between virgin resin and the NOAG-thermoplastic resin compositions is consistent with the conclusion that there is an absence of chemical interaction or reaction between the NOAG and a thermoplastic resin.

- 10 The inventors observed that molded parts consisting of the NOAG-thermoplastic resin composition could be ground and remolded under the same operating parameters.

Detailed analysis of the NOAG-thermoplastic resin indicate that compounding and molding of the NOAG-resin composition do not alter or degrade the resin. For

example, the C<sup>13</sup> nuclear magnetic resonance spectroscopic analysis of the resin before and after molding (curve 61 and curve 59, Figure 14) with NOAG are virtually identical. The similarity of spectra indicates that NOAG does not degrade the resin; if it did, decomposition products with different C<sup>13</sup> spectra should have been detected.

- 5 Their absence indicates no detectable degradation of the resin. Similarly, differential scanning calorimetry analysis indicate that the crystal structure and abundance in PP is indistinguishable from PP molded with 0.5 and 1.0 weight of NOAG (curves 67, 65, and 63 in Figure 15).

In spite of careful and multi-directional research, the inventors have not been  
10 able to detect any discernable change in the base resin composition or structure. Yet, the NOAG-resin composition has important and significant behavioral properties in the thermoplastic molding environment.

#### Benefits Of Using NOAG In A Thermoplastic Molding Process

The benefits of using the new NOAG-thermoplastic resin composition in a  
15 thermoplastic molding process that the inventors have to date identified are:

A. Higher productivity because of:

1. Reduced cycle time – combination of reduced cure time, fill time, pack and hold time.
2. Increased line speeds – extruder revolutions per minute and feet  
20 per minute output.
3. De-molding enhancement articles release more easily from the mold surface.
4. Continuous purging and cleaning.

B. Lower power usage per part because of:

1. Reduced heater demand in barrel and screw.
2. Reduced screw speed.
3. Reduced injection pressure.
4. Less force needed to achieve desired injection speed.
5. Significant reduction in cure and cycle time.

C. Reduced wear on equipment because of:

1. Lower operating temperatures.
2. Lower operating pressures.
3. Reduced screw and injection speeds.

#### Benefits Of Using NOAG-Thermoplastic Resin To Make Products

The articles manufactured from the NOAG-thermoplastic resin composition experience considerable benefits:

- A. Improved fill in complex tooling geometries allowing molding of more complex articles.
- B. Smoother surface finishes.
- C. Reduced sink marks.
- D. Improved dispersion of additives and pigments.
- E. Reduced degradation of polymer due to lower operating temperatures.

These improvements to the manufactured article come with the additional advantage that the article experiences no loss of physical, mechanical or thermal properties as compared to one made from virgin resin.

Advantages To The User Of NOAG-Thermoplastic Compositions

A manufacturer who molds plastic parts will see the following advantages to using NOAG-thermoplastic resin composition because of:

- 5           A.     Lowered manufacturing costs – higher profits.
1.     Higher productivity, increased parts/hour, higher profitability.
2.     Increased capacity.
3.     Reduced energy consumption per part manufactured.
4.     Reduced mold release agents used.
5.     Reduced down time for purging, cleaning, and maintenance.
- 10          B.     Non-specific thermoplastic resin response.
1.     All benefits of the NOAG-resin composition are affective across  
                      the thermoplastic range of resin types.
2.     No loss of physical, mechanical or thermal properties in all  
                      resins tested.
- 15          3.     No detectable polymer degradation products detectable by  $H^1$   
                      and  $C^{13}$  NMR spectroscopy and DSC spectrometry.
- C.     Wide Selection Of Molding Processes.
- Injection, extrusion, blow, extrusion blow, injection blow, blow film  
                  extrusion, calendaring, thermoforming, casting and expansion processes
- 20           can use the NOAG-thermoplastic resin composition to make goods.